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(72) Inventor: Hill, Randal Myron
Midland, Michigan 48642 (US)

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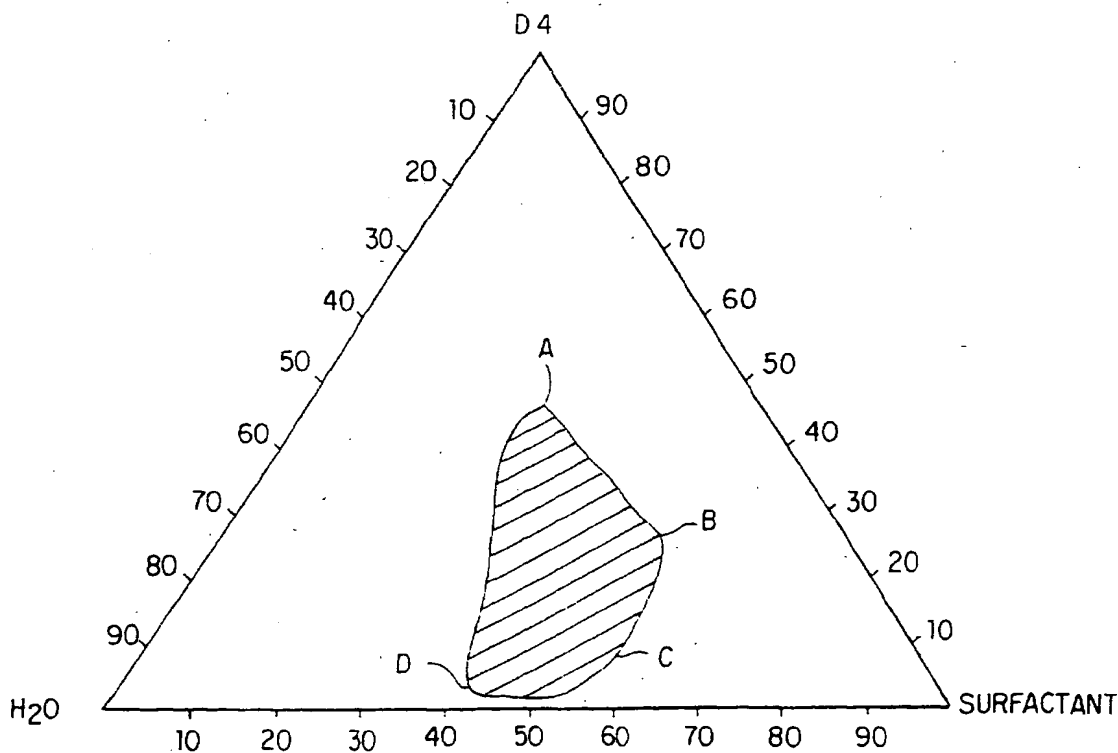
(74) Representative: Kyle, Diana et al
Elkington and Fife
Prospect House
8 Pembroke Road
Sevenoaks, Kent TN13 1XR (GB)

(71) Applicant: DOW CORNING CORPORATION
Midland Michigan 48686-0994 (US)

(54) Clear silicone gels

(57) A method of forming a thermodynamically stable transparent product is disclosed by combining (i) water; (ii) a volatile cyclic methyl siloxane or volatile linear methyl siloxane; and (iii) a silicone polyether surfactant. The amounts of each component, the particular type of

silicone polyether surfactant used and the order of addition of the components, influence the type of product obtained and these factors are such that the composition forms an optically clear gel. The gel is useful in personal care products such as cosmetics and antiperspirants.

**FIG. 1****EP 0 789 061 A1**

Description

This invention is directed to a transparent silicone and, more particularly, to a ternary composition of water, a volatile cyclic or linear methyl siloxane (VMS) and a short-chain or low molecular weight silicone polyether, which when combined provides an optically clear gel.

It is well documented (US-A 4,999,398) that emulsions, especially silicone emulsions, are opaque, cloudy and tend to separate on standing. Thus, silicone microemulsions which contain micro-particles in the droplet phase providing a measure of clarity or which are gels that are thermodynamically stable transparent structures are desirable.

As used herein, the term emulsion or macroemulsion means a dispersion of one immiscible liquid in another, in the form of droplets with diameters approximately in the range of 100-1,000 nanometers (0.1-1.0 microns/1,000-10,000 angstroms A).

In contrast, a microemulsion means a transparent dispersion of two or more immiscible liquids and a surfactant as used herein. Microemulsions are clear or transparent because they contain particles smaller than the wavelength of visible light, which is typically on the order of 10-100 nanometers.

Microemulsions may contain oil droplets dispersed in water (O/W), water droplets dispersed in oil (W/O) or they may be in the form of a bi-continuous structure. They are characterized by an ultra-low interfacial tension between the oil and water phases.

A microemulsion is recognized by several of its inherent characteristics which are that (i) it contains oil, water and a surfactant; (ii) there is a high concentration of surfactant relative to oil; (iii) the system is optically clear; (iv) the phases do not separate by centrifugation; and (v) the system forms spontaneously.

Clarity or transparency is controlled to a great extent by the particle size of the dispersed phase because the scattering of light is dependent on particle size. Therefore, clear or transparent compositions appear to be a single phase without droplets or particles when viewed with the naked eye.

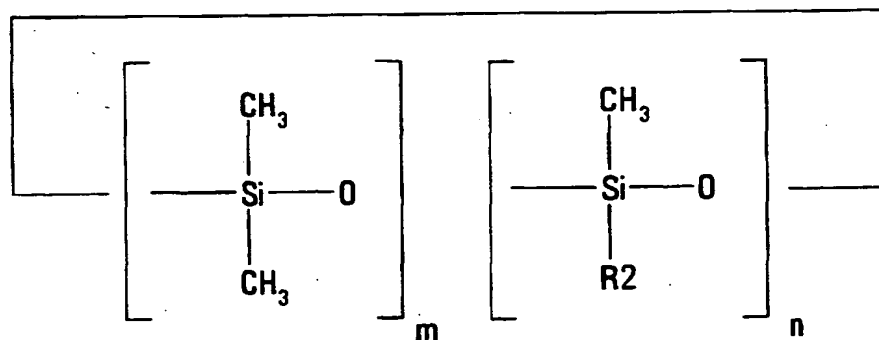
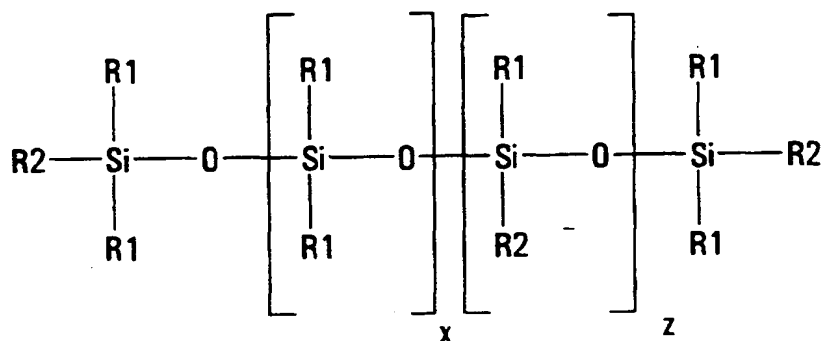
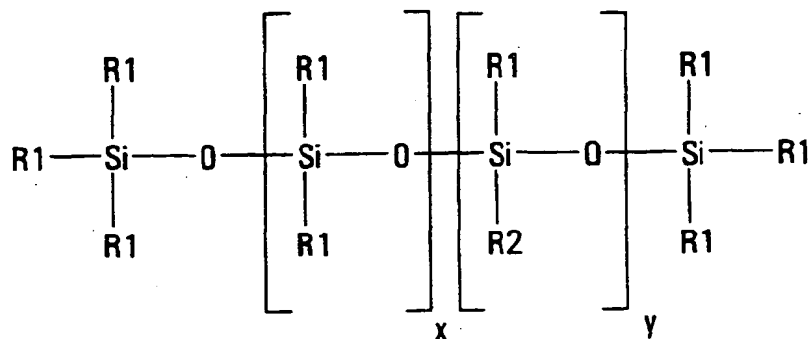
On the other hand, and in contrast to an emulsion or microemulsion, a gel is a semi-solid stabilized or set by a three-dimensional lattice system. Because a surfactant has a bipolar amphiphilic structure (i.e., a water-soluble polar head and a water-insoluble organic tail), surfactant molecules accumulate preferentially at the interface of two immiscible phases. If the concentration at the interface exceeds the critical micelle forming concentration (cmc), a colloidal surfactant solution is produced. With increasing concentration, or volume fraction of the internal phase, such a solution becomes a structured gel.

U.S. 3,299,112 describes products formed from a ternary system of water, a silicone oil and a silicone polyether. But in contrast to our invention, the products in U.S. 3,299,112 are emulsions which are not clear; the ternary system therein is not a gel; the silicone oil is not a volatile cyclic VMS; and the linear silicone oil therein is not a volatile linear VMS.

Rather, the patented silicone oil corresponds to $R^*_3SiO(R^*_2SiO)_xSiR^*_3$ where x is 10-1,000. Our corresponding volatile linear VMS has an "x" of 0-5, well below the above range. In fact, where "x" exceeds 5, the product emulsions tend not to be clear.

Furthermore, emulsions from U.S. 3,299,112 are recognized as inherently unstable systems that separate with time. In contrast, our gels are stable indefinitely. The order of addition of our respective components does influence product formation and with mild mixing at room temperature (20-25°C./68-77°C.), that is sufficient to yield stable gels.

Our method for making a gel composition comprises sequentially (i) combining a silicone polyether and a cyclic methyl siloxane of the formula $\{(CH_3)_2SiO\}_p$ or a linear methyl siloxane of the formula $(CH_3)_3SiO\{(CH_3)_2SiO\}_qSi(CH_3)_3$ in which p is 3-6 and q is 0-5; (ii) mixing the silicone polyether with the cyclic or linear methyl siloxane; (iii) adding water to the mixture of silicone polyether and cyclic or linear methyl siloxane; and (iv) mixing the water, silicone polyether and cyclic or linear methyl siloxane until a clear gel forms; said silicone polyether having a formula selected from the group consisting of



where R¹ is an alkyl group containing 1-6 carbon atoms; R² is $-(\text{CH}_2)_a\text{O}(\text{C}_2\text{H}_4\text{O})_b(\text{C}_3\text{H}_6\text{O})_c\text{R}^3$; x is 0-3; y is 1-3; z is 0-2; m is 3-5; n is at least one; a is 3-6; b is 10-30; c is 0-5; and R³ is hydrogen atom, a methyl radical or an acyl radical.

Our clear gel has particular value in the personal care arena. Because of the unique volatility characteristics of the VMS component of a ternary system, it can be used alone or blended with other cosmetic fluids to form a variety of over-the-counter personal care products.

Thus, it is useful as a carrier in antiperspirants and deodorants, since it leaves a dry feel and does not cool the skin upon evaporation. It is lubricious and will improve the properties of skin creams, skin care lotions, moisturizers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, liquid soaps, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave and after-shave lotions, shaving soaps and shaving lathers. It can also be used in hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories and cuticle coats, to enhance gloss and drying time and to provide conditioning benefits.

In cosmetics, it will function as a leveling and spreading agent for pigments in make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, color cosmetic removers and powders. It is also useful as a delivery system for oil and water soluble substances such as vitamins. When incorporated into sticks,

other gels, lotions, aerosols and roll-ons, our ternary composition imparts a dry, silky-smooth, pay-out.

Additionally, our clear gel exhibits a variety of advantageous and beneficial properties such as (i) clarity, (ii) the ability to combine properties of water and oil in a single homogeneous material, (iii) shelf stability and (iv) ease of preparation. Consequently, it has wide application but is especially in cosmetics or antiperspirants.

A transparent gel for cosmetic or antiperspirant preferably is a semi-solid material at rest, but will thin sufficiently under moderately applied shear stress, to flow easily and smoothly when spread over the skin.

Beyond personal care, the claimed gels have application in formulating consumer products such as ski waxes, insecticides, stain removal sticks, car waxes, tire treatments and vinyl protectants.

It is an object of this invention to form a clear silicone gel by simply combining (i) water; (ii) a volatile cyclic methyl siloxane or volatile linear methyl siloxane; and (iii) a silicone polyether surfactant. What results as a product is significant because we have discovered how to make clear silicone products without involving the use of high shear and without the necessity of adding a separate gelator such as stearyl alcohol, cetyl alcohol or hydrogenated castor oil, to achieve a semi-solid state.

The ternary composition of this invention contains water, a volatile cyclic or linear methyl siloxane and a short-chain or low molecular weight silicone polyether. Those three components are combined to form clear gel compositions without the addition of other materials.

Thus, the composition is free of non-essential ingredients such as gelators; salts; co-surfactants; monohydroxy alcohols; and diols and triols such as ethylene glycol and glycerol. The elimination of such non-essential ingredients is especially beneficial and advantageous, as it obviates the need for refractive index matching, often necessary in the past to achieve clear or transparent products.

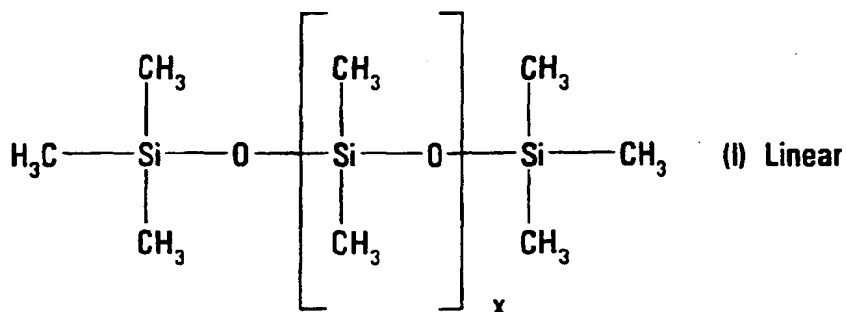
The three components of this ternary system, however, cannot be combined in any given order of addition to easily obtain a gel. Although these gels are manifestations of thermodynamically stable liquid crystal phases, their viscosity is very high and they require unacceptably long periods of time to form spontaneously. Because they are shear thinning, they mix easily if only sufficient shear is applied to break down their gel structure. The amount of shear necessary to do this will be evident from the discussion below in connection with Example II. Thus, it is important that the oil component (i.e., the VMS) and the silicone polyether component be combined in the mixing container before addition of water to the container. The oil component and the silicone polyether component are then mixed together for a short period of time in the mixing container. The third component (i.e., water) is then added to the VMS and the silicone polyether in the mixing container and the three components are mixed in the mixing container for another short period of time.

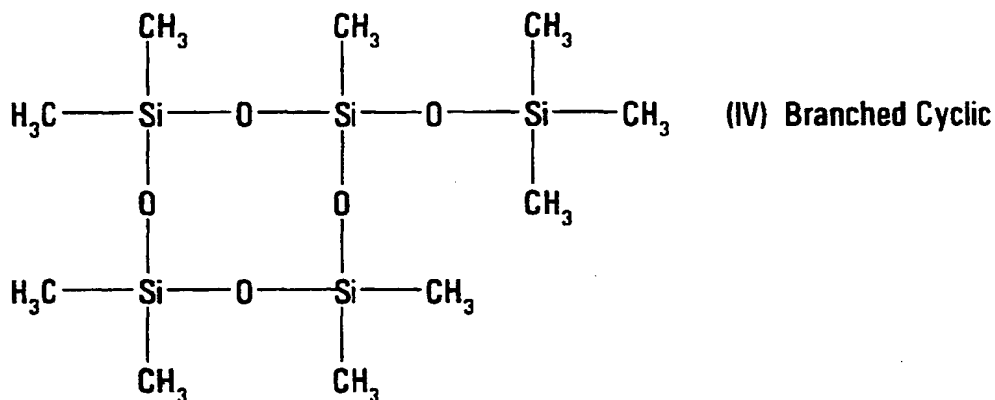
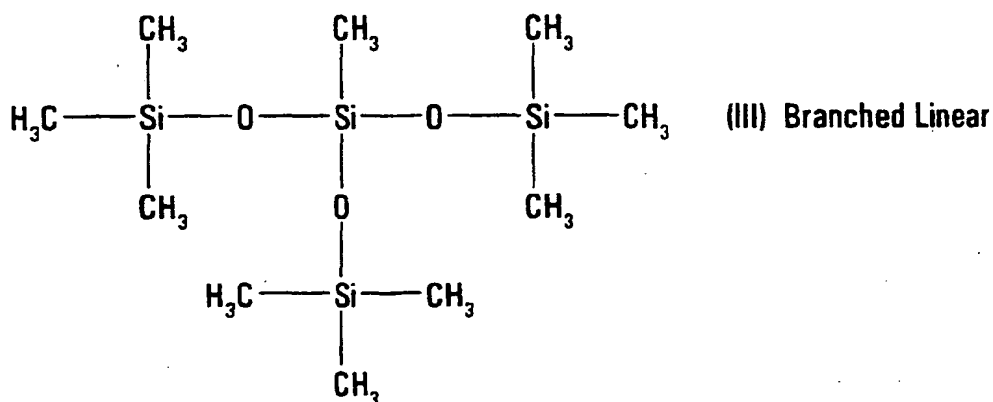
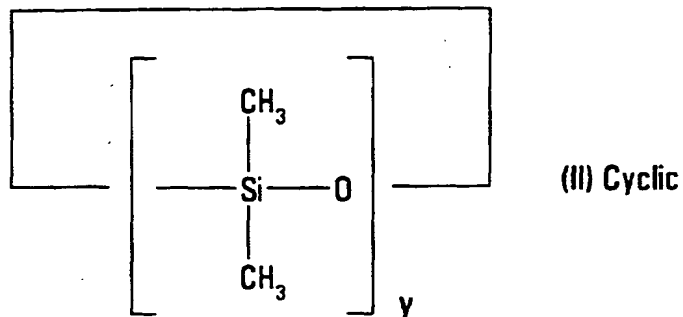
While heat enhances solubility, lowers surface tension and reduces viscosity, its application is not required. Room temperature mixing is sufficient in most cases.

The oil component of our ternary composition, i.e., the VMS, is a low viscosity silicone fluid corresponding to the average unit formula $(\text{CH}_3)_a\text{SiO}_{(4-a)/2}$ in which a has an average value of two to three. The fluid contains siloxane units joined by Si-O-Si bonds. Representative units are monofunctional "M" units $(\text{CH}_3)_3\text{SiO}_{1/2}$ and difunctional "D" units $(\text{CH}_3)_2\text{SiO}_{2/2}$. The presence of trifunctional "T" units $\text{CH}_3\text{SiO}_{3/2}$ results in the formation of branched cyclic VMS. The presence of tetrafunctional "Q" units $\text{SiO}_{4/2}$ results in the formation of branched linear VMS.

Linear VMS have the formula $(\text{CH}_3)_3\text{SiO}\{(\text{CH}_3)_2\text{SiO}\}_x\text{Si}(\text{CH}_3)_3$ and cyclic VMS have the formula $\{(\text{CH}_3)_2\text{SiO}\}_y$, in which x is 0-5 and y is 3-6. Preferably, the VMS has a boiling point less than 250°C . and a viscosity of 0.65-5.0 centistokes (mm^2/s).

Some representative VMS are:





Representative linear VMS (I) are hexamethyldisiloxane (MM) with a boiling point of 100°C., viscosity of 0.65 mm²/s and formula Me₃SiOSiMe₃; octamethyltrisiloxane (MDM) with a boiling point of 152°C., viscosity of 1.04 mm²/s and formula Me₃SiOMe₂SiOSiMe₃; decamethyltetrasiloxane (MD₂M) with a boiling point of 194°C., viscosity of 1.53 mm²/s and formula Me₃SiO(Me₂SiO)₂SiMe₃; dodecamethylpentasiloxane (MD₃M) with a boiling point of 229°C., viscosity of 2.06 mm²/s and formula Me₃SiO(Me₂SiO)₃SiMe₃; tetradecamethylhexasiloxane (MD₄M) with a boiling point of 245°C., viscosity of 2.63 mm²/s and formula Me₃SiO(Me₂SiO)₄SiMe₃; and hexadecamethylheptasiloxane (MD₅M) with a boiling point of 270°C., viscosity of 3.24 mm²/s and formula Me₃SiO(Me₂SiO)₅SiMe₃.

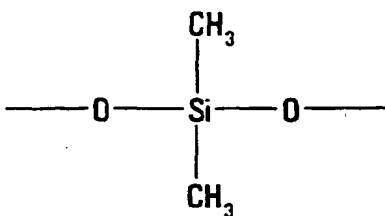
Cyclic VMS (II) have been assigned the International Nomenclature Cosmetic Ingredient (INCI) name "CYCLOMETHICONE" by The Cosmetics, Toiletries and Fragrance Association, Inc., (CTFA) Washington, DC. Cyclic and linear VMS are clear fluids, essentially odorless, non-toxic, non-greasy, non-stinging and non-irritating to skin. VMS leave substantially no residue after 30 minutes at room temperature (20-25°C./68-77°F.) when one gram is placed at the

center of No. 1 circular filter paper of 185 millimeters diameter, supported at its perimeter in open room atmosphere. VMS may be used alone or mixed together in combinations. Mixtures result in solutions having evaporating behaviors different from individual fluids.

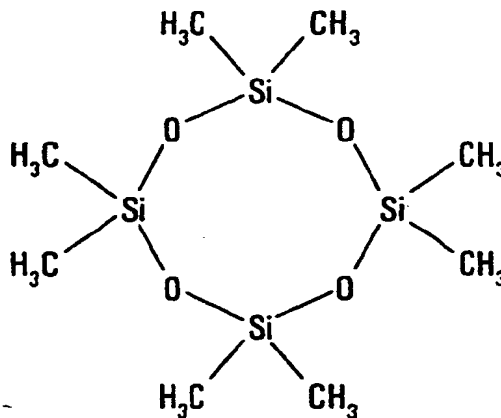
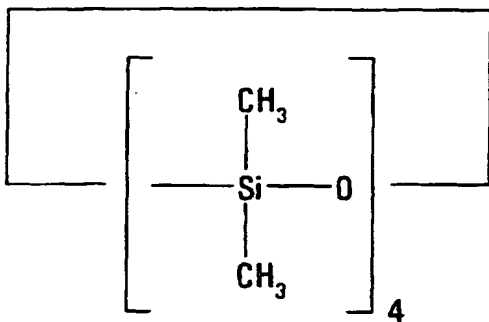
Representative cyclic VMS (II) are hexamethylcyclotrisiloxane (D3) a solid with a boiling point of 134°C. and formula $\{(\text{Me}_2)\text{SiO}\}_3$; octamethylcyclotetrasiloxane (D4) with a boiling point of 176°C., viscosity of 2.3 mm²/s and formula $\{(\text{Me}_2)\text{SiO}\}_4$; decamethylcyclopentasiloxane (D5) with a boiling point of 210°C., viscosity of 3.87 mm²/s and formula $\{(\text{Me}_2)\text{SiO}\}_5$; and dodecamethylcyclohexasiloxane (D6) with a boiling point of 245°C., viscosity of 6.62 mm²/s and formula $\{(\text{Me}_2)\text{SiO}\}_6$.

Representative branched VMS (III) and (IV) are heptamethyl-3-((trimethylsilyl)oxy)trisiloxane (M₃T) with a boiling point of 192°C., viscosity of 1.57 mm²/s and formula C₁₀H₃₀O₃Si₄; hexamethyl-3,3-bis((trimethylsilyl)oxy) trisiloxane (M₄Q) with a boiling point of 222°C., viscosity of 2.86 mm²/s and formula C₁₂H₃₆O₄Si₅; and pentamethyl ((trimethylsilyl)oxy) cyclotrisiloxane (MD₃) with the formula C₈H₂₄O₄Si₄.

One preferred VMS component of our ternary system is octamethylcyclotetrasiloxane $\{(\text{CH}_3)_2\text{SiO}\}_4$. It has a viscosity of 2.3 centistokes (mm²/s) at 25°C. and is referred to as "D₄" since it contains four difunctional "D" units $(\text{CH}_3)_2\text{SiO}_{2/2}$, i.e.,

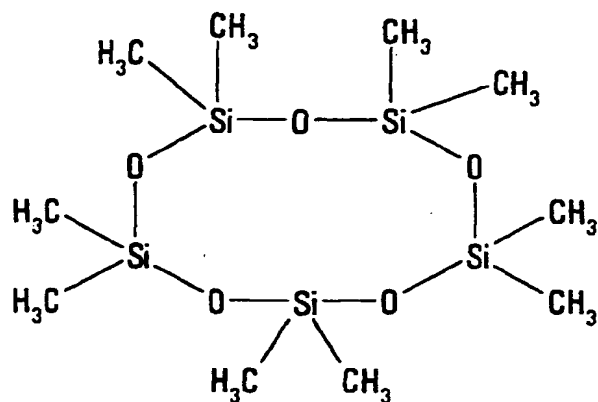


Four "D" units combine to form octamethylcyclotetrasiloxane which is shown by either structure below:

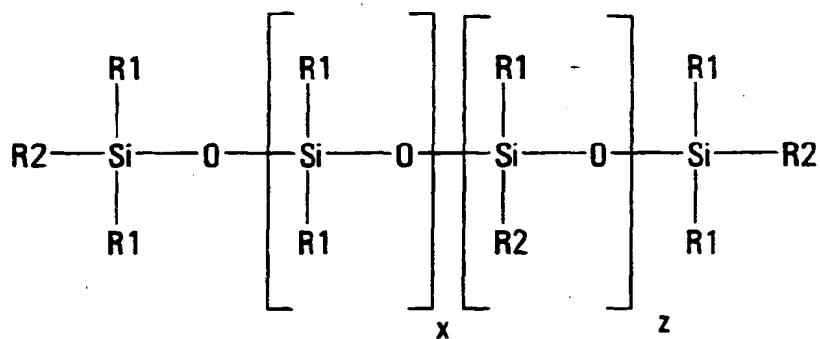
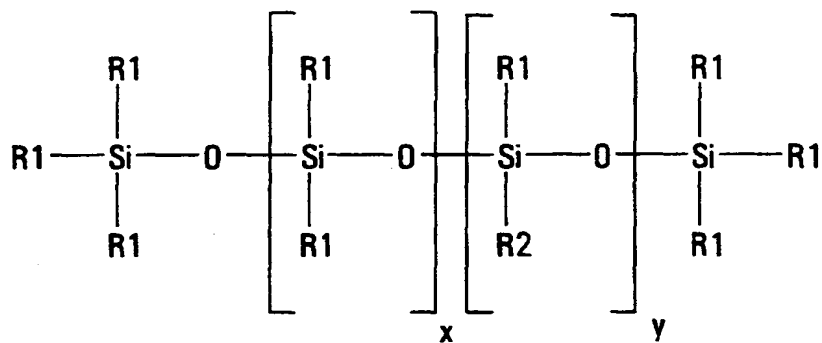


D4 often referred to as TETRAMER has a higher viscosity (2.3 mm²/s) and is thicker than water (1.0 mm²/s), yet D4 needs 94% less heat to evaporate than water.

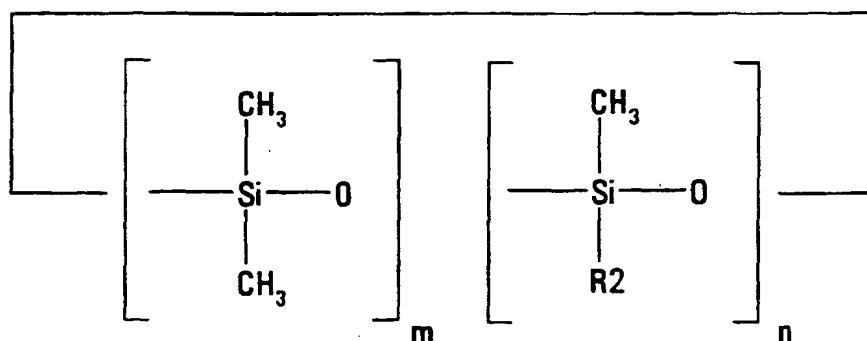
Another preferred VMS component of our ternary system is decamethylcyclopentasiloxane (D5), often referred to as PENTAMER, and shown below:



The other component of this ternary system, in addition to water and VMS, is a short-chain or low molecular weight silicone polyether. Representative linear polyethers are:



Cyclic polyethers as below can also be used.



In these structures, R¹ is an alkyl group containing 1-6 carbon atoms such as methyl, ethyl, propyl, butyl, pentyl and hexyl; R² is the radical $-(\text{CH}_2)_a\text{O}(\text{C}_2\text{H}_4\text{O})_b(\text{C}_3\text{H}_6\text{O})_c\text{R}^3$; x is 0-3; y is 1-3; z is 0-2; m is 3-5; n is at least one; a is 3-6; b is 10-30; c is 0-5; and R³ is hydrogen atom, a methyl radical or an acyl radical such as acetyl. Preferably, R¹ is methyl; x is zero; y is one; z is one; n is one; b is 13-24; c is zero; and R³ is hydrogen. Most preferably, b is 16-20.

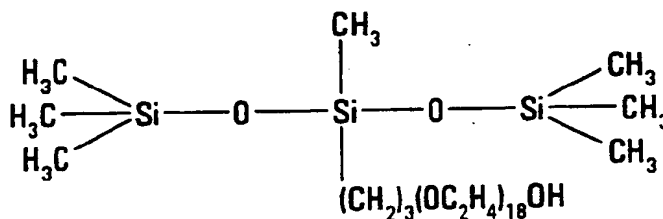
While the compositions of this invention may contain 5-70% by weight of surfactant, most preferably they contain 15-30% by weight of surfactant. The remainder of the composition is the VMS oil component and water, with the proportions of VMS and water generally falling between 40:60 to 80:20.

For purposes of the invention, the criteria used to determine optical clarity is whether text can be read with the naked eye through a two centimeter diameter bottle filled with the gel.

This is a reliable and legitimate technique for evaluating gels and microemulsions. For example, as noted in the textbook *Microemulsions Theory and Practice*, Edited by Leon M. Prince, Academic Press, Inc., Pages 7-10, New York (1977), the "Visual recognition of microemulsions should not be taken lightly. In fact, the microemulsion chemist should train himself carefully in this art. Use of sunlight rather than an artificial source of light is recommended. The eye is better than a microscope because the limit of resolution of a light microscope in blue light is only about 0.1 μm so that droplets smaller than 0.14 μm cannot be seen".

Example I

An optically clear gel at room temperature was formed by first adding 70 parts of octamethylcyclotetrasiloxane (D4) to a mixing bowl. Then, 45 parts of silicone polyether was added to the bowl. These two components were mixed together in the mixing bowl for ten seconds using a single speed HAUSCHILD DENTAL MIXER. Next, 45 parts of de-ionized water were added to the bowl. The three components were then mixed together in the mixing bowl for another ten seconds using the aforementioned mixer. The solution gelled and was clear. The text was legible through a two centimeter diameter bottle filled with the gel. The silicone polyether used in this and the following examples was the trisiloxane:



The viscosity and shear thinning properties were similar to those obtained in Example II.

Figure 1 is a ternary phase diagram of a system comprising water, octamethylcyclotetrasiloxane (D4) and the silicone polyether surfactant shown in Example I, at 25°C; for determining composition ranges of gels prepared according to my invention. Gel compositions are defined by the shaded area ABCD, i.e., the gel region.

In Figure 1, each of the corners represents 100 percent of the component labeled there. The side of the triangle directly opposite each corner represents zero percent of that component. Lines parallel to the opposite side represent increasing amounts of that component as they become closer to the corner.

For example, any line drawn from the corner of the component D4 to the opposite side represents varying the amount of D4 at a constant ratio with respect to the other two components H₂O and SURFACTANT.

The composition of any point within the shaded area ABCD is determined by drawing lines parallel to each of the three sides through the point. The amount of each component is then read from the intersection of each line with the side of the triangle which corresponds to that component, i.e. the side beginning at 100 at each component's corner.

Technically, shaded area ABCD represents the I₁ or the cubic phase liquid crystal region. The remaining area of the triangle outside of shaded area ABCD contains other regions (not shown), such as regions of unstable emulsions, microemulsions L, lamellar phase liquid crystals L_α and hexagonal phase liquid crystals H₁.

Example II

Example I was repeated and formed an optically clear gel at room temperature by first adding 60 parts of decamethylcyclopentasiloxane (D5) to a mixing bowl. 45 parts of silicone polyether were then added to the mixing bowl. These two components were mixed together in the mixing bowl for ten seconds using the above HAUSCHILD™ MIXER. Next, 46 parts of de-ionized water were added to the mixing bowl. The three components were then mixed together in the mixing bowl for another ten seconds using the same HAUSCHILD™ MIXER. The solution gelled and was clear. The optical clarity was the same as obtained in Example I.

The flow properties of the gel formed in this example were examined using a Carri-Med™ rheometer, in which the viscosity was determined at linearly increasing values of shear stress ranging from 0 to 17,500 dyne/cm². At a low applied shear stress of 2,500 dyne/cm² or less, which corresponds to rest conditions, the measured viscosity was 700,000 poise (70 million centipoise/70 million mPa·s). This value was found to fall smoothly to less than 10,000 poise (1 million centipoise/1 million mPa·s) at an applied shear stress of 15,000 dyne/cm².

Example III

Example I was again repeated and formed an optically clear gel at room temperature by first adding 60 parts of D4 to a mixing bowl. 45 parts of silicone polyether were then added to the mixing bowl. These two components were mixed together in the mixing bowl for ten seconds using the HAUSCHILD™ MIXER. Next, 49 parts of de-ionized water were added to the mixing bowl. The three components were then mixed together in the mixing bowl for another ten seconds using the above HAUSCHILD™ MIXER. The solution gelled and was clear. The optical clarity was the same as obtained in Example I. The viscosity and shear thinning properties were similar to those obtained in Example II.

The following example illustrates preparation of a gel composition according to my invention using a linear volatile methyl siloxane (VMS) instead of a cyclic VMS.

Example IV

Example I was repeated and formed an optically clear gel at room temperature by first adding 60 parts of dodecamethylpentasiloxane (MD₃M) to a mixing bowl. 45 parts of silicone polyether were then added to the mixing bowl. These two components were mixed together in the mixing bowl for ten seconds using the above HAUSCHILD™ MIXER. Next, 52 parts of de-ionized water were added to the mixing bowl. The three components were then mixed together in the mixing bowl for another ten seconds using the same HAUSCHILD™ MIXER. The solution gelled and was clear. The optical clarity was the same as obtained in Example I. The viscosity and shear thinning properties were similar to those obtained in Example II.

The following two examples illustrate preparation of clear gel antiperspirants. In Examples V and VI, an antiperspirant salt was incorporated into our clear silicone gel.

Example V

Example I was repeated and formed an optically clear gel at room temperature by first adding 61 parts of D4 to a mixing bowl. 46 parts of silicone polyether were then added to the mixing bowl. These two components were mixed together in the mixing bowl for ten seconds using the above HAUSCHILD™ MIXER. Next, 60 parts of an aqueous solution containing 10% of the antiperspirant active Aluminum Chlorohydrate (ACH) were added to the mixing bowl. The components were then mixed together in the mixing bowl for another ten seconds using the same HAUSCHILD™ MIXER. The solution gelled and was clear. The optical clarity was the same as obtained in Example I. The viscosity and shear thinning properties were similar to those obtained in Example II.

Example VI

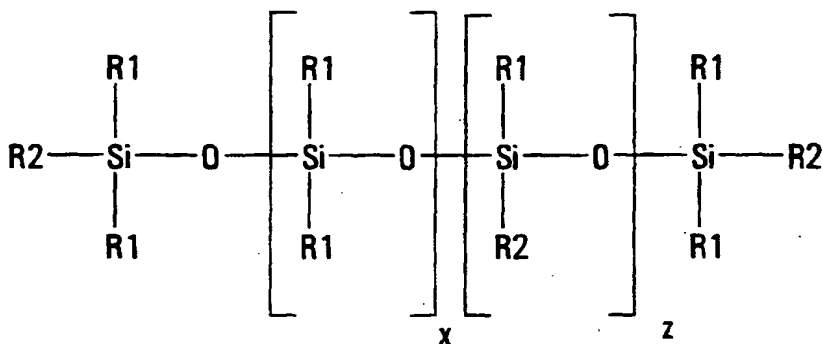
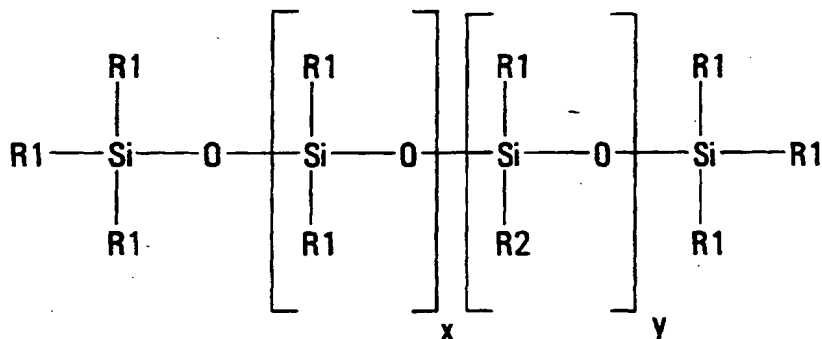
Example I was repeated and formed an optically clear gel at room temperature by first adding 60 parts of D4 to a mixing bowl. 46 parts of silicone polyether were then added to the mixing bowl. These two components were mixed together in the mixing bowl for ten seconds using the above HAUSCHILD™ MIXER. Next, 69 parts of an aqueous solution containing 20% of the antiperspirant active ACH were added to the mixing bowl. The components were then mixed together in the mixing bowl for another ten seconds using the same HAUSCHILD™ MIXER. The solution gelled and was clear. The optical clarity was the same as obtained in Example I. The viscosity and shear thinning properties were similar to those obtained in Example II.

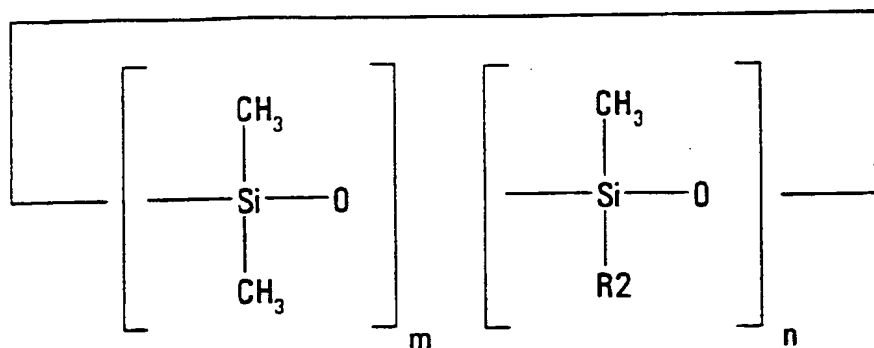
Other antiperspirant salts could have been used in Examples V and VI such as Aluminum Dichlorohydrate, Aluminum Sesquichlorohydrate, Aluminum-Zirconium Trichlorohydrex-Gly (AZG), Aluminum-Zirconium Tetrachlorohydrex-Gly, Aluminum-Zirconium Pentachlorohydrex-Gly and Aluminum-Zirconium Octachlorohydrex-Gly. Any formulated antiperspirant product should contain a maximum use level of antiperspirant salt active of 20% by weight AZG-type and 25% by weight ACH-type on an anhydrous basis.

Gels prepared according to Examples I-VI were semi-solid materials at rest, which shear thinned upon being subjected to moderately applied shear stress.

Claims

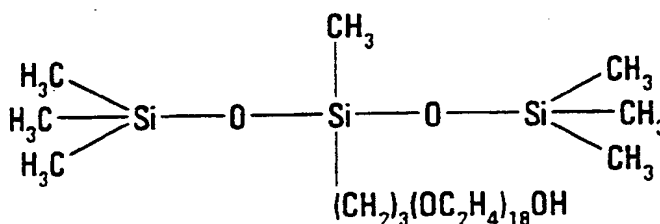
1. A method of making a gel composition comprising sequentially (i) combining a silicone polyether and a cyclic methyl siloxane of the formula $\{(\text{CH}_3)_2\text{SiO}\}_p$ or a linear methyl siloxane of the formula $(\text{CH}_3)_3\text{SiO}\{(\text{CH}_3)_2\text{SiO}\}_q\text{Si}(\text{CH}_3)_3$ in which p is 3-6 and q is 0-5; (ii) mixing the silicone polyether with the cyclic or linear methyl siloxane; (iii) adding water to the mixture of silicone polyether and cyclic or linear methyl siloxane; and (iv) mixing the water, silicone polyether, and cyclic or linear methyl siloxane until a clear gel forms; the silicone polyether having a formula selected from the group consisting of



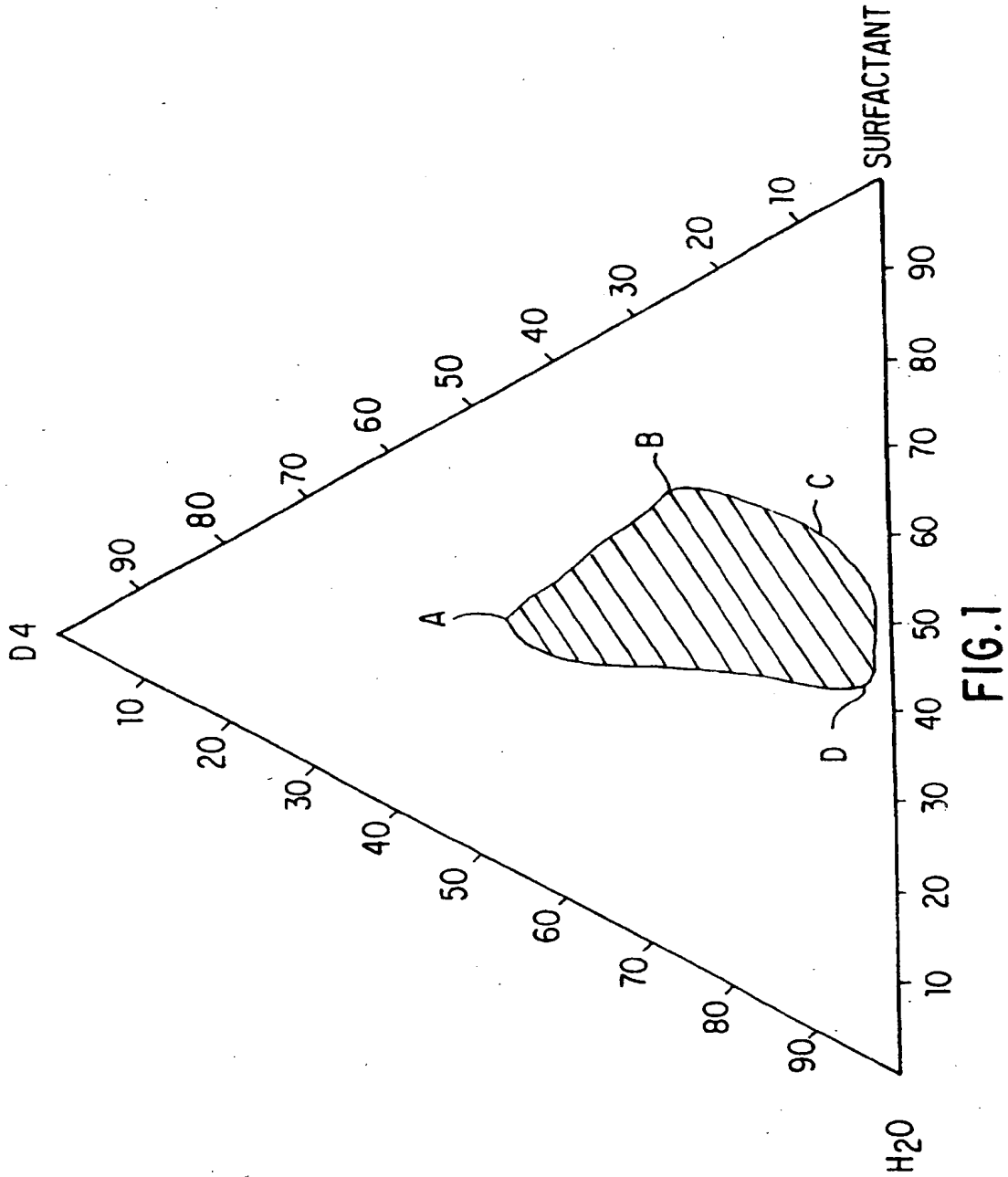


where R1 is an alkyl group containing 1-6 carbon atoms; R2 is $-(\text{CH}_2)_a\text{O}(\text{C}_2\text{H}_4\text{O})_b(\text{C}_3\text{H}_6\text{O})_c\text{R3}$; x is 0-3; y is 1-3; z is 0-2; m is 3-5; n is at least one; a is 3-6; b is 10-30; c is 0-5; and R3 is hydrogen atom, a methyl radical or an acyl radical.

2. A method according to claim 1 in which the methyl siloxane is selected from octamethylcyclotetrasiloxane or decamethylcyclopentasiloxane.
3. A method according to claim 1 in which the silicone polyether is the compound



4. A gel obtainable by the method according to claim 2, the gel having a composition defined by and within the shaded area depicted in Figure 1.
5. A gel obtainable by the method according to claim 1 or 2 containing 15-30% by weight of the silicone polyether, the remainder being methyl siloxane and water in proportions between 40:60 to 80:20.
6. A gel obtainable by the method according to claim 1 or 2 having a viscosity of at least 70,000 Pa-s (700,000 poise) at an applied shear stress of 2,500 dyne/cm² which decreases to less than 1,000 Pa-s (10,000 poise) at an applied shear stress of 15,000 dyne/cm².
7. A personal care product containing the gel obtainable by the method according to claim 1 or 2 selected from the group consisting of antiperspirants, deodorants, skin creams, skin care lotions, moisturizers, acne removers, wrinkle removers, facial cleansers, liquid soaps, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave lotions, after-shave lotions, shaving soaps, shaving lathers, hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, cuticle coats, make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, cosmetic removers, delivery systems for oil and water soluble substances and powders; including products in the form of sticks, gels, lotions, aerosols and roll-ons.
8. Use of the personal care product of claim 7 for application to hair or skin.



EP 0 789 061 A1

European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 97 30 0724

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
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A	US 5 456 906 A (POWELL VIRGINIA V ET AL) 10 October 1995 * column 1, line 28 - column 3, line 64; claims; examples *	1,2,4,7,8	
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A,D	US 3 299 112 A (UNION CARBIDE CORP.) 17 January 1967 * the whole document *	1	
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The present search report has been drawn up for all claims			
Place of search MUNICH		Date of completion of the search 7 May 1997	Examiner Hutton, D
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document I : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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